ANTIMONY (ATOMIC ABSORPTION, DIRECT ASPIRATION)

1.0 SCOPE AND APPLICATION

1.1 See Section 1.0 of Method 7000

2.0 SUMMARY OF METHOD

2.1 See Section 2.0 of Method 7000.

3.0 INTERFERENCES

3.1 See Section 3.0 of Method 7000 if interferences are suspected.

3.2 In the presence of lead (1,000 mg/L), a spectral interference may occur at the 217.6-nm resonance line. In this case, the 231.1-nm antimony line should be used.

3.3 Increasing the acid concentrations decreases the antimony absorption. To avoid this effect, the acid concentration in the samples and in the standards should be matched.

3.4 Excess concentrations of copper and nickel (and possibly other elements), as well as acids, can interfere with antimony analyses. If the sample contains these matrix types, either matrices of the standards should be matched to those of the sample or the sample should be analyzed using a nitrous oxide/acetylene flame.

4.0 APPARATUS AND MATERIALS

4.1 For basic apparatus, see Section 4.0 of Method 7000.

4.2 Instrument parameters (general):

4.2.1 **Antimony hollow cathode lamp or electrodeless discharge lamp.**
4.2.2 **Wavelength:** 217.6 nm (primary); 231.1 nm (secondary).
4.2.3 **Fuel:** Acetylene.
4.2.4 **Oxidant:** Air.
4.2.5 **Type of flame:** Fuel lean.
4.2.6 **Background correction:** Required.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000.

5.2 Preparation of standards:

5.2.1 **Stock solution:** Carefully weigh 2.7426 g of antimony potassium tartrate, $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$ (analytical reagent grade), and dissolve in Type II water. Dilute to 1 liter with Type II water; 1 mL = 1 mg Sb (1,000 mg/L). Alternatively, procure a certified standard from a supplier and verify by comparison with a second standard.

5.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should contain 0.2% (v/v) HNO_3 and 1-2% v/v HCl, prepared using the same types of acid and at the same concentrations as in the sample after processing.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chapter Three, Section 3.1.3, Sample Handling and Preservation.

7.0 PROCEDURE

7.1 Sample preparation: The procedures for preparation of the sample are given in Method 3005. Method 3005, a soft digestion, is presently the only digestion procedure recommended for Sb. It yields better recoveries than either Method 3010 or Method 3050. There is no hard digestion for Sb at this time.

7.2 See Method 7000, Paragraph 7.2, Direct Aspiration Procedure.

8.0 QUALITY CONTROL

8.1 See Section 8.0 of Method 7000.

9.0 METHOD PERFORMANCE

9.1 The performance characteristics for an aqueous sample free of interferences are:

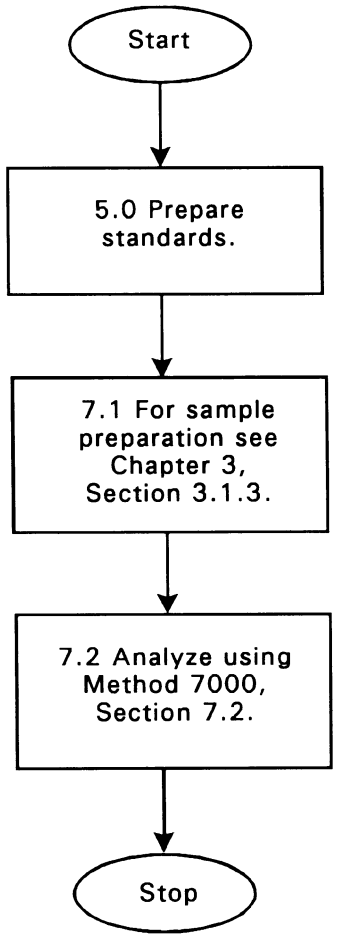
Optimum concentration range: 1-40 mg/L with a wavelength of 217.6 nm.
Sensitivity: 0.5 mg/L.
Detection limit: 0.2 mg/L.

9.2 In a single laboratory, analysis of a mixed industrial-domestic waste effluent, digested with Method 3010, at concentrations of 5.0 and 15 mg Sb/L gave the standard deviations of $\pm$0.08 and $\pm$0.1, respectively. Recoveries at these levels were 96% and 97%, respectively.

9.3 For concentrations of antimony below 0.35 mg/L, the furnace procedure (Method 7041) is recommended.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 204.1.


Start
5.0 Prepare standards.
7.1 For sample preparation see Chapter 3, Section 3.1.3.
7.2 Analyze using Method 7000, Section 7.2.
Stop